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Surface Roughness and Wear Behavior of Occlusal Splint Materials Made of Contemporary and High-Performance Polymers

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Abstract

With the development of a digital technology of computer assisted manufacturing (CAD/CAM) and new age materials, the use of new types of occlusal splint is to consider. The aim of the present study was to evaluate the surface roughness (Ra) and wear behavior of different CAD/CAM materials against enamel antagonist through a simulated chewing test. A total of 75 specimens made from Ethylene Vinyl Acetate (EVA), Polymethyl methacrylate (PMMA), Polycarbonate (PC), Polyetheretherketone (PEEK), and Polyethyleneterephthalate (PETG) as a control were polished to evaluate the Ra before loading by optical profilometry and further analyzed by scanning electron microscopy (SEM). Specimens of each group were subjected to thermomechanical fatigue loading in a chewing simulator (60000 cycles at 49 N with 5°C-55°C thermocycling). The wear volume loss and change in Ra of each specimen after the simulated chewing were analyzed. One-Way ANOVA, paired samples t-test and Pearson correlation analysis were performed for statistical analyzes. The result showed that the volume loss and Ra varied among the materials tested. EVA exhibited the greatest amount of Ra and volume loss ($p<0.001$), while PEEK had the lowest values for both ($p<0.001$). In terms of volume loss, there was no significant difference between PC and PMMA ($p>0.05$). SEM investigations revealed different wear behaviors, especially in EVA. As PEEK showed significantly more favorable results, PEEK splints should be considered as a new therapeutic option for occlusal splint.

Key words Occlusal splint ● PEEK ● Wear ● Surface Roughness ● EVA

Introduction

Temporomandibular disorders (TMD) have multiple and broad clinical effects on orofacial structures and are often diagnosed in patients with a prevalence between 5% and 12% [1-3]. Among treatment options, occlusal splints, also known as oral appliances, oral orthotics, and bite guards, are the most common therapeutic procedures (68%) and allow interesting clinical outcomes, as they were shown to reduce 70% to 90% of the TMD symptoms successfully [2-7].

Occlusal splints are clinical tools applied on all or most of the teeth in one dental arch and are available in a wide range of designs and materials [4]. These appliances are conventionally fabricated with polyethylene (PVAc-PE), acrylic resin and polymethylmethacrylate (PMMA) by an analog workflow including refractory cast [8-12]. Although, these materials display interesting properties, they are not considered ideal due to potential thermal irritation unpleasant taste, dimensional changes, time consuming process, residual monomers, unfavorable shape or color, susceptibility to fracture, increased risk of denture-associated infections, and skin or respiratory allergies which can affect both patients and dental technicians [12-17]. Besides, in the case of bruxism or parafunction, these materials tend to wear over time, even when the splint is used for a short period of time [11,13]. Wear prevents occlusal contacts from being in equilibrium which is important for a successful splint therapy and reduces the longevity of appliance [5,18]. This wear pattern is dramatically important for serious clenching activities that can induce complex deformations of paradental tissues, like condyles, rami and dental arches [19]. Therefore, such effects have a negative impact on treatment outcomes and patient compliance which are accepted as crucial for the success of the treatment, emphasizing the need for more adapted materials [20].

Accordingly, digital dentistry has open new area of research and development to overcome these limitations [21]. Digital occlusal splints have been reported to have advantages over conventional ones due to superior materials and fabrication methods [22]. Computer-aided design/computer-assisted manufacturing (CAD/CAM) systems allow splints to be made from prefabricated and standard materials, such as polycarbonate (PC) and PMMA discs [8,21,23,24]. These materials are considered as interesting options for occlusal splints, as they are high-performance-polymers with less susceptibility to fracture, reducing individual human errors during technical processes, and exhibiting superior material properties to those of conventional ones [21,25-29]. Another alternative for the fabrication of occlusal splints may be polyetheretherketone (PEEK). PEEK is a tooth colored polymeric material and has been tested for prosthodontic applications such as removable and fixed prostheses [30,31].

Although PEEK is a promising material in the dental field and suggested as a possible occlusal splint material, there are no available data allowing to validate such hypothesis [32-34].

To fill this gap of knowledge, the aim of this study was to compare the surface roughness, wear volume loss, and wear behavior of five commercially available materials by generating two-body wear process through chewing simulation and evaluate PEEK as a new option for occlusal splint.

Materials and methods

Types of tested materials

Five different types of resin materials were evaluated in this study. Detailed information about type, manufacturer and basic composition of materials tested in this study is presented in **Table 1.**

Preparation of specimens

Cylindrical specimens (thickness: 2 mm, diameter:10 mm) of each material were designed by a universal CAD software (inLab SW 4.2.1; Sirona Dental Systems, NY, USA) [5,35]. The designed specimens were milled from selected materials by using the subtractive method (inLab MC X5; Sirona Dental Systems, NY, USA). Before milling process of the materials, the equipment was calibrated to minimize errors. All specimens were finalized with a stepwise polishing with the use of discs of grain sizes 2500 and 4000 grit on a rotary machine under wet conditions (Buehler Metaserv Motopol 12; Buehler, Coventry, Great Britain). Then, the specimens were washed in an ultrasonic bath (ZOKOP 6L; Zokop, Glendale, CA, USA) for 10 min at room temperature and embedded into acrylic molds (Duracryl Self-cure; Erk Dental, İzmir, Turkey).

Surface roughness measurement after polishing

After polishing, surface roughness (Ra) of all the specimens was determined by a three-dimensional non-contact profilometer (AEP Nanomap-1000WLI; AEP TECHNOLOGY, Santa Clara, CA, USA) in order to avoid surface damaging. Profiles of 9 mm^2 surface located in the different areas of the specimens were measured with an optical resolution of 550 nm. Average of Ra values were calculated with SPIP software (Image Metrology A/S, Lyngby, Denmark) by using at least three measurement results, according to ISO 4287 [36].

In addition, scanning electron microscope (SEM) (Carl Zeiss EVO LS 10; Carl Zeiss NTS, Germany) (10000x magnification) was used to identify surface alterations and porosities for each material. Samples were coated with Au-Pd prior to analysis in order to prevent charging and at least three images were taken for each sample.

Wear test with chewing simulation

Two body wear of the specimens was conducted using the chewing simulator machine (MOD Chewing Simulator; MOD Dental, Ankara, Turkey) which can test 6 antagonists and abrader simultaneously **(Figure 1).** The chewing simulator was equipped with an enamel antagonist to mimic the oral conditions during bruxism. Enamel antagonists were prepared from caries-free extracted human maxillary molars donated by anonymous patients and the Ethics Committee of the Istanbul University Faculty of Dentistry approved this study under protocol number 2019/19. All teeth were cleaned of both soft tissues and calculus and stored in 0.1% thymol solution (Thymol, Supelco®, Sigma-Aldrich Chemie GmbH, St. Louis, Missouri, USA) at room temperature afterward. Root portions of them were positioned for the sample surfaces with a custom-made paralleling machine and fixed inside plastic rings (Ø 36 mm) by embedding in autopolymerizing acrylic resin material (Duracryl Self-cure; Erk Dental, İzmir, Turkey). Standardization of the enamel antagonists for shape and size was carried out by drilling cusp portions in a cupola-like contour by using concave drills having grain sizes of 40 µm and 8µm (Frank Dental GmbH, Gmund am Tegernsee, Germany).

Each chamber of the chewing simulator consisted of an upper antagonist and lower specimen holder which were fixed to the chamber with screws. The parameters of the chewing simulation used for the present study were shown in **Table 2**. The simulator was programmed to provide cyclic loading and reciprocating movement to achieve masticatory pattern. Chewing simulation was performed with a 60 seconds dwell time and 0.8 Hz frequency. Enamel antagonists achieved a vertical movement of 5 mm and a descending speed of 55 mm/s to stroke specimen surfaces with a horizontal movement of 2 mm. The vertical load value was maintained at 5 kg during the motion as being equivalent to 49 N of effective masticating force which is used as a standard [37]. Additionally, included thermocycling system was utilized during wear simulation under the condition of 5-55°C with a heating and cooling system by a programmable logic. Each specimen was tested for 60000 cycles to simulate approximately three months of clinical service for occlusal splint [38].

Surface roughness and volume loss measurements after wear testing

Three-dimensional surface profile images of all specimens were created, and visual analysis of the images was performed with the Average Ra values were calculated. To measure volume loss after chewing simulation, all images were imported to a reverse engineering software (RapidForm XOR3; GeomagicInc, Cary, NC, USA) which combines images into solid work for measurements. Volume loss estimation for each sample was performed by subtracting the volume of solid meshbox of worn specimen from the total volume.

Statistical analysis

For each tested material, a sample size of 12 in each group was estimated with $\alpha = 5\%$ and 90% power. Fifteen specimens per group were analyzed considering possible damage or technical problem. Statistical analyses were performed using statistical software (SPSS V23; IBM Corp. Armonk, NY). All data were submitted to Shapiro-Wilk test to test the normality of data, and One-way ANOVA was used to compare normal distribution data between groups. The differences between groups were determined by using a Tamhane's T2 from multiple comparison tests. Paired samples t-test was used to compare the Ra values for each material group separately before and after chewing simulation. The relationship between increase in surface Ra and volume loss were determined by Pearson correlation analysis. Analysis results were presented as means and standard deviation ($\alpha =$ 5%) and a p-value<0.05 was considered significant.

Results

Surface roughness

To evaluate the wear of each tested materials following chewing simulation, surface roughness was measured using the same three-dimensional non-contact profilometer that was used before the chewing test **(Table 3).**

The Ra values before chewing simulation were significantly different among tested materials (p<0.001). EVA group showed the greatest surface roughness (0.235 ± 0.026 µm), while the PEEK group exhibited the lowest values (0.139 ± 0.017 µm). After chewing simulation, significant differences were observed between groups (p<0.001). As observed before chewing simulation, EVA displayed the greatest Ra values (3.879 ± 0.4 µm), while PEEK had the lowest ones (0.889 ± 0.138 µm).

Interestingly, chewing simulation induced a significant increase of Ra for all tested materials. The greatest amount of increase in surface roughness was shown for EVA (3.644 ± 0.42 µm), and the lowest ones were belonged to PEEK (0.749 ± 0.134 µm), similar to Ra comparison.

Two-body wear of materials tested in chewing simulation

The mean volume loss (mm^3) and increase in surface roughness(µm), which are important indicators of the wear rate of materials, after 60000 cycles were presented in **Table 4**. As a result of wear process, the 3D surface images and profile curves of EVA, PC and C groups showed rougher surfaces compared to PEEK and PMMA groups after chewing simulation **(Figure 2)**.

It was observed that the increased values vary according to the groups (p<0.001). While the greatest change in Ra was measured in the EVA group, the lowest change was obtained in PEEK group. There was a significant difference between the groups for the mean values of volume loss (p<0,001). The highest mean value for the volume loss was in the EVA, while the lowest mean value was obtained for PEEK. Also, for PC, a slight negative correlation between the increase in surface roughness and the volume loss was detected (r= -0.635; p=0.027).

SEM evaluation and surface profiles

SEM representative images and optical profilometry of the tested materials before and after chewing simulation are described in **Figure 3.**

Before wear test, all specimen surfaces appeared relatively uniform and smooth, also having appropriate Ra values by not exceeding a threshold of 0.2 µm [39]. Regarding morphological observations of worn surfaces after chewing, SEM images of the specimens displayed visual cues for volume loss such as irregularities, pits, valleys, scratches and inhomogeneities. EVA displayed the most irregular surface among all groups by forming several big defects with ruptures, debris caused by partial spallation and wear, and revealing particles protruding from the surface, apparently dislodged from matrix of the material. It was also noticed that the mass of wear traces which coalesced each other was distributed over the roughening surfaces and some EVA particles which did not fall off remained on the specimen surface. In the images of PC and C groups, fine flaws like cracks, surface irregularities, bulges, scratches and shallow defects were observed, and some small valleys were also identified. The surfaces of the wear areas of PMMA and PEEK in contact with the molar tooth revealed relatively smooth surfaces only with tiny valleys and pits.

Discussion

In this study, changes in surface roughness and wear volume loss of different materials against the human maxillary molars with 2-body wear simulation were investigated. The overall results of this study showed a correlation between the increase in Ra values and volume loss of the groups after chewing simulation, while volume loss values were equal for two groups (PC and PMMA); thus, the main null hypothesis tested in the present study was partially rejected. The second null hypothesis that PEEK would be a good alternative occlusal splint material was accepted according to the tested parameters.

The wear resistance and wear behavior are of paramount importance as occlusal loads during parafunction, especially bruxism, can occur higher than 785 N [40]. The relationship between volume loss and changes in surface roughness which is directly proportional to the rate of wear was also evaluated [41]. Surface roughness is affected by clinical adjustments like polishing, as polishing leads to smooth surfaces that undergo less wear and provides the advantage of extended longevity of the restoration [42-44]. Thus, polishing is recommended to prevent occlusal splint surfaces from being worn and to achieve optimal clinical performance. The results of wear and Ra for tested materials were expected and explained by having more increase in surface roughness, revealing more volume loss. This statement is supported by the SEM images taken from the different specimens. The difference in wear is most probably due to the variations in mechanical properties of the tested materials, which might result in different degrees of wear. To our knowledge, this is the first study that evaluated all types of present and possible splint materials together, so comparison has been performed partially with literature due to limited data. In previous studies, PC has been found to be superior to PMMA and resin materials in terms of wear and roughness. These results slightly differ from the present study, as we have found wear volume loss of PC and PMMA was equal [24,45]. The negative correlation between the increase in surface roughness and volume loss for PC may be explained by the surface hardness value or the wear behavior of the material used [46]. The discrepancies between the results can be due to the different types of PMMA materials, fabrication process (milled, injectable, and conventional), antagonist and wear test settings. Because, hydrothermal aging (performed in the present study) and water absorption have been found to alter wear behavior previously [47]. Additionally, due to their physical properties, conventional materials tend to absorb more water compared to CAD/CAM materials [48]. A negative correlation between the increase in surface roughness and volume loss for PC may be explained by the surface hardness value or the wear behavior of the material used [46].

Another study which evaluated PMMA and resin materials showed the highest material wear volume loss for resins, followed by milled and conventional PMMA. According to applied test wear test settings, our results have been found to exhibit more wear than this study. At 60000 wear cycles PMMA in our study showed 2.182 ± 0.11 mm^3 volume loss, while the PMMA of the above-mentioned study had 1.8 ±0.4 mm^3 at 120000 cycles [49]. The difference in volume loss between studies can be best explained by the included hydrothermal aging in our study and type of antagonist, as these researchers did not apply hydrothermal aging and used only mesiobuccal cusp of a molar tooth as antagonist. The change of temperature between 5°C and 55° for 60000 cycles during the chewing simulation might lead to thermal expansion and shrinkage of the polymer groups. This might have accelerated their fatigue during wear procedure, resulting in significant wear pattern. In a recent study, polyamide and different fabricated PMMA materials were tested as occlusal splint materials and revealed best wear results for polyamide. As no polyamide group was included in the present study, it was not possible to make an exact comparison, but milled PMMA may be implemented to use as a splint material with satisfactory results, consistent with our results [50].To the authors' knowledge, the only comparable study using EVA for occlusal devices was carried out by Pena et al., as this material is not so popular for occlusal splints. In that study, the mechanical behaviors of EVA were evaluated and good results in force dissipation were reported. The authors recommended EVA as a splint material due to its shock-absorbing capacity, low cost and easy handling [51]. However, EVA wore out the most among all groups in the present study and had the highest Ra values after an approximate application time comparable to 3 months of clinical usage with wear simulation, contrasting with the results from Pena *et al*. Thus, the long-term use of this material should not be recommended for long splint therapies or can be used only for short term therapies.

PEEK is biocompatible and was recommended as a promising dental material for long term restorations [52]. Due to its physical and mechanical properties similar to dentin and bone, it has a variety of dental applications from implantology to orthodontics [33,53]. PEEK has a lower Young's (elastic) modulus (3-4 GPa) than dentin (elastic modulus: 15 GPa), but it is possible to increase this value up to 18 GPa by modifying PEEK and incorporating other materials [54,55]. Considering lower elastic modulus, PEEK is expected to cause less antagonist wear, as observed in a recent study that evaluated antagonistic primary tooth wear, but this was not evaluated in the present study [56]. Additionally, it has high fracture resistance and abrasive properties [57,58]. Despite low hardness and elastic modulus, PEEK has been shown to have competitive abrasive resistance with metallic alloys [58]. Consisting with the results of the study by Wimmer et al, PEEK was found to have a reduced volume loss after wear test among all the tested resin materials in the present study [59]. Although, high-performance polyetherketoneketone (PEKK) was used in another study, greatest material wear was found in PEKK when compared to ceramics and PMMA-based CAD/CAM materials with similar wear patterns, contrasting with the results of this study [56]. This discrepancy can be attributed to three factors: type of preferred materials, antagonist tooth, and performed wear cycle in simulation. In terms of chair-side modifications or clinical adjustments of PEEK, it is possible to condition the PEEK surface to facilitate its bonding with provisional resin and composite resin by using air abrasion, silica coating, adhesive systems containing MMA-monomers, and etching with various acids like sulphuric and piranha. PEEK is also another option which can be applied an alternative to other CAD/CAM materials like PMMA for dental restorations by having the advantage of being digitally fabricating [33]. However, it is more expensive than other splint materials, and this can be the most important disadvantage of the PEEK. However, our results support the fact that PEEK may be a safe material as an occlusal splint due to its physical properties with low risk at antagonist tooth or restoration wear or fracture [56].

The quantitative Ra and wear data measured corresponded to the qualitative investigation by SEM shown in **Figure 1** that exhibit different surface appearances. Before the chewing simulation, all groups showed clinically acceptable Ra values by not exceeding 0.2 μm (allowable limit value of Ra for hard surfaces in the oral environment), except EVA (0.235±0.026 μm) [60,61]. After the chewing simulation, EVA again revealed the highest Ra value (3.879±0.400 μm), besides SEM images of EVA illustrated wide wear areas and deep defects, different from the other materials, and was the weakest candidate as a splint material among all. PC and C groups presented scratches and cracks, while PEEK and PMMA look smoothly abraded. According to these images, wear behaviors of the groups had different characterization against the load supporting the results of the study by Prpic et al [62]. 2D-3D surface appearances of the worn materials and depicted surface profile schemes in **Figure 2** are also in accordance with the SEM images. Taken together, the importance of our results is that the surface appearance of the images shows a relation to the wear volume loss which apparently depends on the composition and characterization of the material having different cross-link densities and conversion degrees suggesting that wear behavior dominates over the differences between the materials. Modern technologies allow us to produce CAD/CAM splint materials under standardized polymerization conditions and eliminate the polymerization shrinkage, resulting in extreme homogeneity, greater accuracy, less wear, favorable esthetic, greater long-term stability, better biocompatibility and improved wearing comfort [23,26].

Limitations of this study that may affect the clinical interpretation of the present results included the difficulty in replicating clinical conditions, using water rather than artificial saliva during wear test, evaluating only volume loss of the tested materials not also the likely effect on the opposing teeth, and using one type antagonist. Clinical studies should be performed to validate the obtained results in the present study and future researches are needed to have wear phenomena as in the masticatory system, performing the fluctuating pattern of bruxism with varying chewing forces, increasing the number of chewing cycles and evaluating the wear behavior of antagonists.

Conclusion

Considering the aspects mentioned above, this study indicates that PC and PMMA exhibit less wear as occlusal splint materials than those from EVA and C, with the best results for PEEK. It should be noted that when the rough surface of the occlusal splint is detected, the application of polishing should be carried out to prevent increased wear. Additionally, dental practitioners should consider these differences when choosing a material for occlusal splint and be careful about repetitive wear facets after clinical adjustment. Taken together, 'mix splint' that has specific wear-resistant areas according to the needs of the patient might be a future reliable clinical option.

Conflict of Interest

The authors declare that they have no conflict of interest.

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Captions to legends:

Tables:

Table 1 List of materials tested.

Table 2 The parameters of chewing simulation.

Table 3 Comparison of mean surface roughness values of tested materials before and after chewing simulation.

Table 4 One-way ANOVA results of the groups for the volume loss and the increase in surface roughness after chewing simulation.

Figures:

Fig.1 **(a)** MOD Chewing Simulator; **(b)** Set up of enamel antagonist and test specimen; **(c)** Implementation of wear test.

Fig. 2 Optical profilometry images showing the 2D (A), 3D (B) surface topography and the profile of roughness (C) (for the line drawn with blue) or worn samples after chewing simulation.

Fig. 3 SEM images of the group samples before and after the wear test. **(a)** EVA (Ethylene Vinyl Acetate); **(b)** C (PETG- Polyethyleneterephthalate); **(c)** PC (Polycarbonate); **(d)** PEEK (Polyetheretherketone), **(e)** PMMA (Polymethyl methacrylate) (magnification 10000x)

Tables:

Trade name	***Type***	***Abbreviation***	***Main composition***	***Batch number/Color***	***Manufacturer***
EVA sheet	Round plate of Ethylene Vinyl Acetate	EVA	Ethylene Vinyl Acetate	3185.1(colorless)	Bioplast®. ScheuTM-Dental. Iserlohn. German
Ceramill PEEK 71	Acrylic, polyetheretherketone	PEEK	Polyetheretherketone (100%)	1105403-760390 (beige)	Amann Girrbach AG, Koblach, Austria
Splint Plus BioStar	Polycarbonate disc	PC	Polycarbonate (100%), 98,5x20mm	650126 (transparent)	ERNST HINRICHS GmbH, Germany
Ceramill A-Splint	Acrylic, polymethylmethacrylate disc	PMMA	PMMA (polymethyl methacrylate)	2305503 - 340345(transparent)	Amann Girrbach AG, Koblach, Austria
Erkodur Disc (Control)	Thermoforming discs	C	PETG (polyethyleneterephthalate - glycol modified / Ethylene - 1,4-cycloexylene dimethylene terephthalate Copolymer)	70-1065-ERK0044 (clear transparent)	ERKODENT Erich Kopp GmbH, Pfalzgrafenweiler, Germany

Table 1 List of materials tested*.

* Written followed by the information of the manufacturers.

Parameter	Characteristics
Sample quantity tested	75
Weight per sample	5 kg
Number of cycles	60000
Cycle frequency	0.8 Hz
Vertical movement	5 mm
Horizontal movement	2 mm
Rising speed	55 mm/s
Descending speed	55 mm/s
Forward speed	55 mm/s
Backward speed	55 mm/s
Hot/cold bath temperature	5°C-55°C
Dwell time	60 s

Table 2 The parameters of chewing simulation.

Group	Before chewing simulation(µm)	After chewing simulation(µm)	*p***
Eva	0.235 ± 0.026c	3.879 ± 0.400a	<0.001
Control	0.203 ± 0.009b	2.140 ± 0.216b	<0.001
PC	0.167 ± 0.025a	1.617 ± 0.286c	<0.001
PMMA	0.192 ± 0.018ab	1.154 ± 0.139d	<0.001
Peek	0.139 ± 0.017d	0.889 ± 0.138e	<0.001
p*	<0.001	<0.001	

Table 3 Comparison of mean surface roughness values of tested materials before and after chewing simulation. a-e: Identical letters indicate no significant differences in the same time period (*p*>0.05), * One-Way ANOVA, **Paired samples t-Test.

Group	Increase in surface roughness(µm)	Volume Loss(mm^3)	Correlation
Eva	3.644 ± 0.42a	3.733 ± 0.448a	r= -0.117; *p*=0.718
Control	1.937 ± 0.216b	3.079 ± 0.164b	r= -0.226; *p*=0.481
PC	1.45 ± 0.288c	2.493 ± 0.42c	**r= -0.635; *p*=0.027**
PMMA	0.962 ± 0.139d	2.182 ± 0.11c	r= 0.184; *p*=0.567
Peek	0.749 ± 0.134e	1.084 ± 0.109d	r= 0.127; *p*=0.694
p*	<0.001	<0.001	

Table 4 One-way ANOVA results of the groups for the volume loss and the increase in surface roughness after chewing simulation. a-e: Identical letters indicate no significant differences in the same time period (p>0.05), r: Pearson's correlation coefficient, * One-Way ANOVA.

Figures:

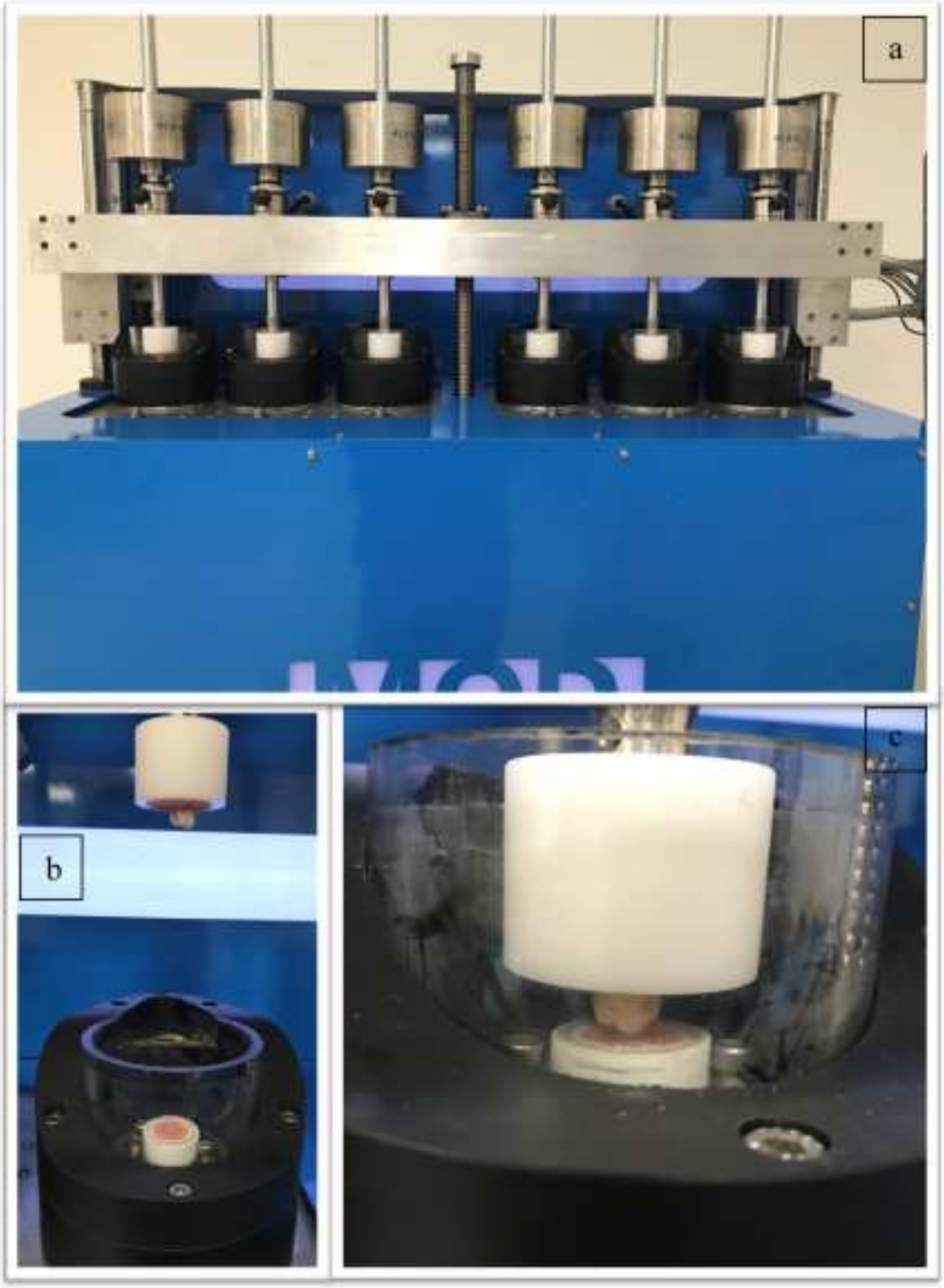
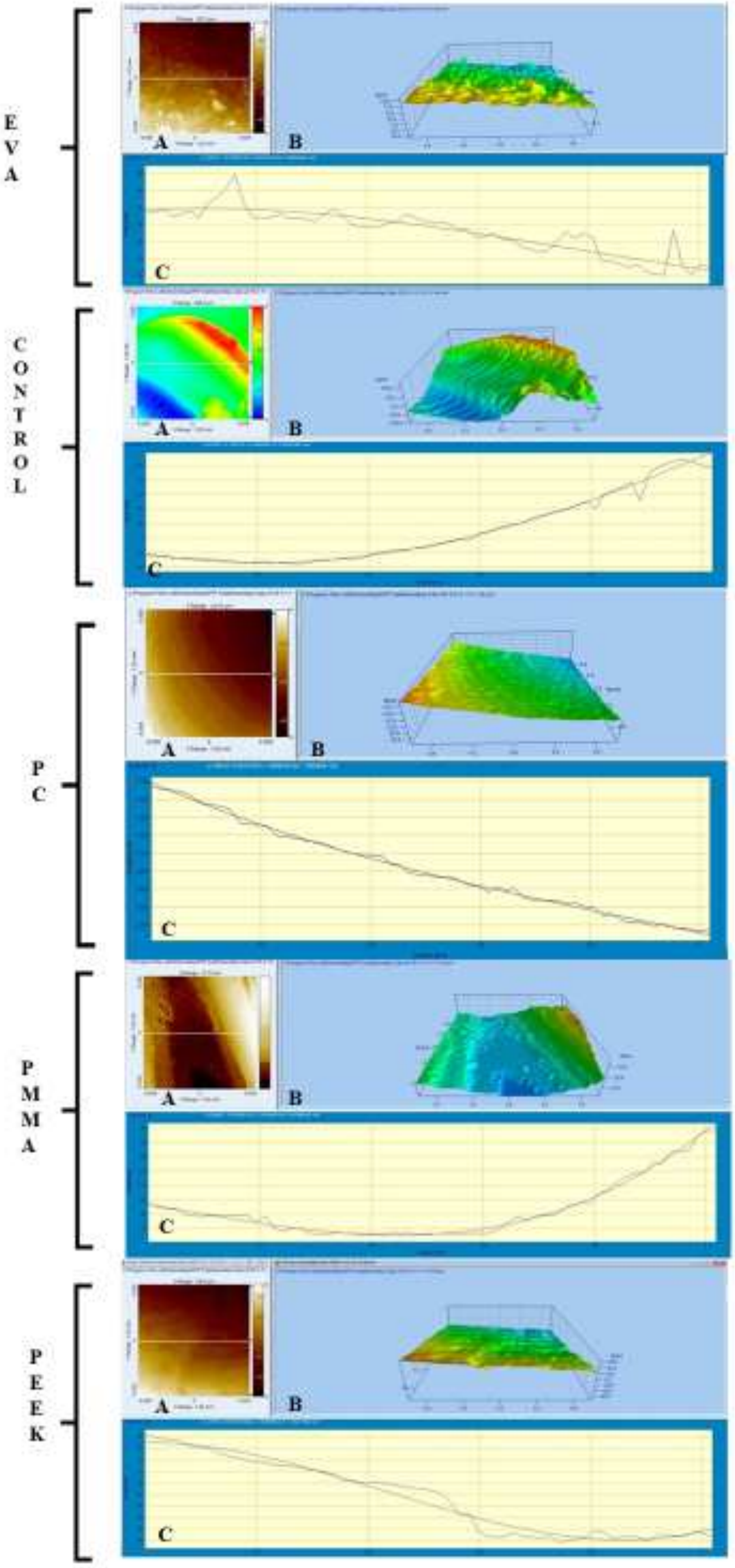


Figure 1.(a) MOD Chewing Simulator; **(b)** Set up of enamel antagonist and test specimen; **(c)** Implementation of wear test.



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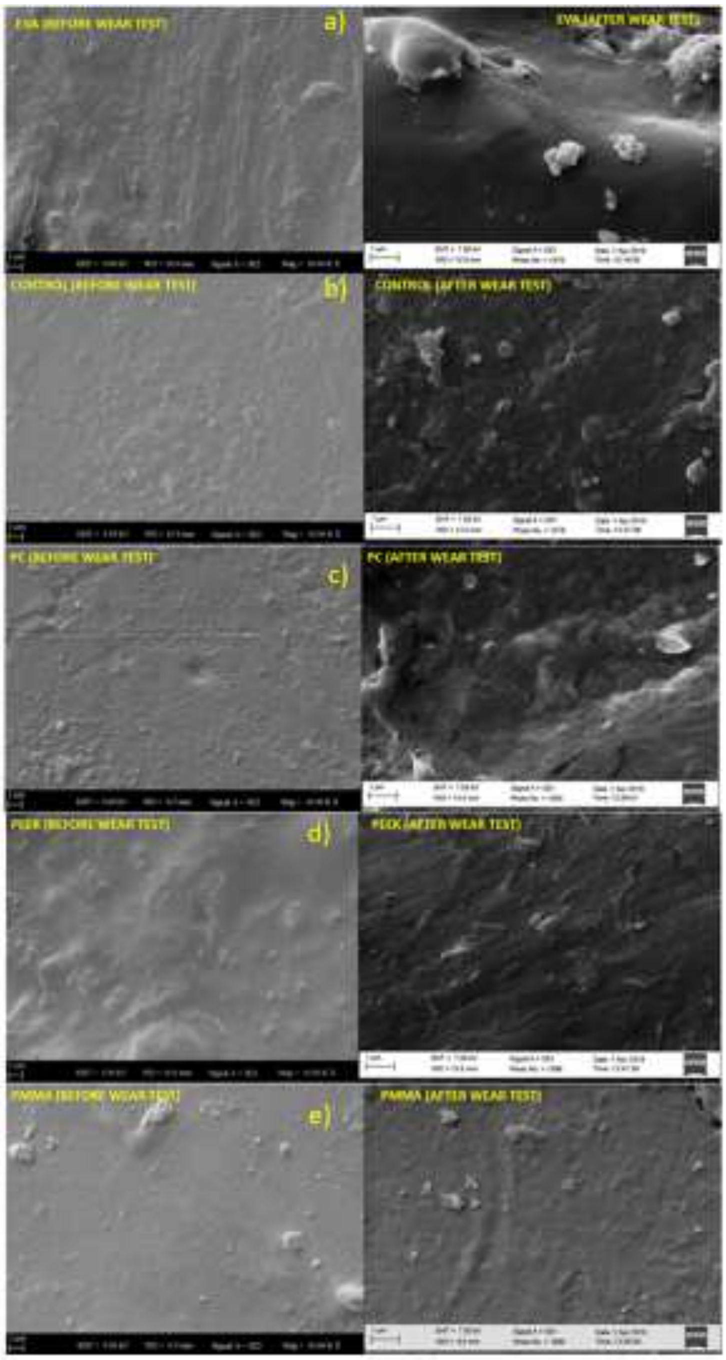

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